

Docket No. 743414-15

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re Patent Application of:)
John Scheirs et al.) Group Art Unit: 1733
Application No. 10/828,504) Examiner: Jessica Rossi
Filed: April 21, 2004) Confirmation No. 4310
For: LAMINATED GLASS) Date: August 1, 2005

SUBMISSION OF CERTIFIED COPY OF PRIORITY DOCUMENT
IN SUPPORT OF CLAIM FOR CONVENTION PRIORITY

Commissioner for Patents
P.O. Box 1450
Alexandria, VA 22313-1450

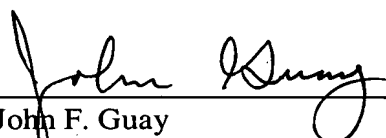
Sir:

The benefit of the filing date of the following prior foreign application in the following foreign country is hereby requested, and the right of priority provided in 35 U.S.C. § 119 is hereby claimed:

<u>Application No.</u>	<u>Country</u>	<u>Filed</u>
2002-952196	AUSTRALIA	October 22, 2002

In support of this claim, enclosed is a certified copy of said prior foreign application. Acknowledgment of receipt of this certified copy is requested.

Respectfully submitted,



John F. Guay
Registration No. 47,248

NIXON PEABODY LLP
Suite 900, 401 9th Street, N.W.
Washington, D.C. 20004-2128
(202) 585-8000

BEST AVAILABLE COPY



Australian Government

Patent Office
Canberra

I, JANENE PEISKER, TEAM LEADER EXAMINATION SUPPORT AND SALES hereby certify that annexed is a true copy of the Provisional specification in connection with Application No. 2002952196 for a patent by OPACI LAM PTY LTD as filed on 22 October 2002.



WITNESS my hand this
Twenty-seventh day of June 2005

A handwritten signature in dark ink, appearing to read 'J. Peisker'.

JANENE PEISKER
TEAM LEADER EXAMINATION
SUPPORT AND SALES

CERTIFIED COPY OF
PRIORITY DOCUMENT

BEST AVAILABLE COPY

A U S T R A L I A

Patents Act 1990

PROVISIONAL SPECIFICATION

for the invention entitled:

"Laminated glass"

The invention is described in the following statement:

LAMINATED GLASS

The present invention relates to laminated glass. In particular, the present invention relates to laminated glass comprising an interlayer comprised of polyvinylchloride (PVC), and to
5 a method of preparing the same.

Laminated glass has many applications as a construction material. It can for example be used to form windows, walls, doors, screens and splashbacks. It can also be used for the construction of articles such as coffee tables, and other small articles of furniture.
10 Laminated safety glass is specified or desired in a number of applications as a result of its inherent safety characteristics compared to normal glass sheets. Laminated glass is commonly formed by joining two sheets of glass using a polyvinyl butyral (PVB) interlayer. The production process normally involves two steps. In one step the interlayer is placed between two sheets of glass and the resulting sandwich type structure is heated as
15 it passes through nip rollers that squeeze the glass sheets into contact with the softened interlayer. A further step involves the treatment of the laminate so formed in an autoclave to complete the bonding process.

A key function of an interlayer within a laminated glass structure is to prevent or at least
20 minimise dispersion of dangerous glass fragments should the glass break. Desirable properties of the interlayer include the following: sufficient shear and tear strength to prevent or at least minimise rupture of the interlayer by the broken glass; sufficient adhesion to the glass to prevent or at least minimise dispersion of the broken glass, acceptable thermal stability and acceptable weatherability.

25

In many applications it is desirable to colour the laminated glass, and in some applications render it translucent or opaque. The colouring can be for the purposes of decoration or can serve a functional purpose by partially or completely obscuring the view through the glass sheet. As used throughout this specification the term "colour" refers to either a solid
30 colour or a substantially solid colour that is textured or patterned, for example to have a wood grain, marble onyx or other effect including effects obtained using interference

pigments and pearl pigments.

Previous attempts to produce coloured laminated glass have involved painting or screen printing the finished product. These techniques do not provide a solid or completely uniform colour which makes the appearance unsatisfactory, especially in applications with backlighting. Other attempts to produce coloured laminated glass have involved introducing a coloured acetate film into the laminate usually between two sheets of interlayer. The coloured laminated glass produced in this way suffers from the disadvantage of lack of uniformity of colour. Furthermore, the acetate layer may deleteriously affect the integrity of bond strength between layers within the laminated glass.

Films made from plasticised PVB are considered to be particularly suited for use as an interlayer in laminated glass products due to their ability to strongly adhere with glass, their transparency and their sound physical and mechanical properties over a broad range of temperatures. However, there are several disadvantages to using plasticised PVB films as an interlayer. A major disadvantage is that such films are relatively expensive. PVB films are also hygroscopic and must be handled under controlled atmospheric conditions to prevent excessive moisture uptake. Furthermore, plasticised PVB film is often subject to blocking problems at the time of windup after film formation due its very tacky surface. To prevent blocking, plasticised PVB films are typically provided with some form of parting means when stored or transported in the form of stacks of die-cut blanks or in the form of rolls. The use of PVB as an interlayer material therefore adds considerable expense to the final laminated glass product.

A further disadvantage of using PVB as an interlayer material is that the polymer itself is not particularly well suited to pigmentation or decoration.

PVC has been considered as a potential replacement for PVB as an interlayer material. PVC is a commercial polymer that is readily available and relatively inexpensive. PVC can be easily calendered to a range of thicknesses, it can be plasticised to give a range of

- 3 -

hardness values, it has suitable physical and mechanical properties for use as an interlayer and it can be pigmented or decorated to give a wide range of colours. In addition, PVC is not water sensitive and unlike PVB, does not require storage in a special controlled humidity environment. However, the use of PVC in laminated glass structures has been
5 limited due to the polymer having practically no inherent ability to adhere to glass.

Attempts have been made to promote adhesion between glass and PVC within laminated glass structures. For instance, US 4,277,583 and US 6,180,246 disclose the use of organosilane compounds as adhesion promoters. Such compounds are typically coated on
10 the surface of the PVC film or dispersed within the PVC film itself. Although the adhesion promoters do initiate a bond between the glass and the PVC, there are many practical problems associated with the effective use of such compounds. For example, additional manufacturing steps are often required in order to apply the technology. Furthermore, the process of achieving an appropriate bond between the PVC and the glass is subject to
15 many complex variables such as surface energy of the PVC film, stability of the silane during lamination and the concentration of the silane available at the surface of the PVC film.

US 4,600,627 discloses a laminated glass structure wherein an ethylene vinylacetate (EVA) polymer is used to bond a PVC interlayer to the glass sheets. However, it has been
20 reported that silane adhesion promoters are required to be used in conjunction with EVA in order to obtain adequate bond strength.

Accordingly, there remains an opportunity to design and develop a laminated glass that
25 meets acceptable quality standards and which can be prepared in an effective and efficient manner using PVC as an interlayer material.

In one aspect, the present invention provides a laminated glass comprising at least two glass sheets, an interlayer comprising polyvinylchloride located between the at least two
30 glass sheets, and a polyurethane layer located between the interlayer and each of the at least two glass sheets.

In another aspect, the present invention provides a method of preparing a laminated glass comprising locating an interlayer comprising polyvinylchloride between at least two sheets of glass, and using a polyurethane layer located between the interlayer and each of the at least two glass sheets to bond the interlayer between the at least two glass sheets.

5

In a further aspect, the present invention provides an interlayer comprising polyvinylchloride having a polyurethane layer disposed on each side of the interlayer.

Surprisingly, it has been found that polyurethane can be used in an advantageously robust and simple manufacturing process to bond an interlayer comprising PVC to glass sheets to form a laminated glass structure which has excellent performance characteristics. Furthermore, by using an interlayer comprising coloured PVC, the laminated glass product in accordance with the present invention may be provided in a variety colours.

10 The polyurethane layer in accordance with the present invention bonds the interlayer between the glass sheets. Preferably, the polyurethane layer is used to adhere a single interlayer directly to the glass sheets. In this case, the glass laminate may be viewed as a structure comprised of five layers, that is glass/polyurethane layer/interlayer/polyurethane layer/glass.

20

Alternatively, multiple interlayers may be used. For example a glass laminate may be formed using two interlayers resulting in a 7 layer structure, that is glass/polyurethane layer/interlayer 1/polyurethane layer/interlayer 2/polyurethane layer/glass. In this case, the glass laminate can be designed to display a different colour from opposed faces by using different coloured interlayers.

25

Accordingly, in yet a further aspect the present invention provides a laminated glass comprising at least two glass sheets, at least two interlayers comprising polyvinylchloride located between each of the at least two sheets, and a polyurethane layer located between adjacent interlays and also between the interlayers adjacent each of the at least two glass sheets.

30

- 5 -

The use of multiple interlayers can also extend to structures where glass layers are placed between the interlayers. For example, a 9 layer structure such as glass/polyurethane layer/interlayer 1/polyurethane layer/glass/polyurethane layer/interlayer 2/polyurethane layer/glass. Laminated glass structures of this type have the added advantage of exhibiting
5 improved impact resistance.

In accordance with the present invention, the polyurethane is preferably a thermoplastic polyurethane. In particular, the polyurethane is preferably an aliphatic polyurethane, more preferably an aliphatic polyester-polyurethane.

10

Polyurethanes have been found to be particularly effective at forming a strong bond between glass and the interlayer within the laminated structure, and if more than one interlayer is present, between the interlayer's themselves. In particular, interfacial shear strength values derived from glass laminates of the present invention have been found to
15 be equal or better than those derived from conventional PVB glass laminates. Preferably, the bond between the respective layers has sufficient strength to transfer loads placed on the glass to the interlayer without delamination or ingress of air occurring. It is believed that the silanol (-Si-OH) rich surface of glass and the polar pendant chloride groups residing on the surface of interlayer from the PVC interact with the polyurethane to form a
20 strong bond through hydrogen bonding. For this reason, hydroxy functionalised polyurethanes are preferred.

A particularly preferred aliphatic polyester polyurethane comprises a hexane diol adipate polyurethane, such as that sold by Stahl USA under the product name of RU-41-347.
25 Preferably, the polyurethane is waterborne, has a viscosity from about 5 to about 250 cps and a solids content from about 30 weight % to about 45 weight %, more preferably about 39 weight %. The polyurethane preferably has a tensile strength from about 1800 psi to about 2200 psi, more preferably about 2000 psi, an elongation from about 300% to 350 %, more preferably 325 %, a modulus at 100 % from about 1100 psi to about 1300 psi, more
30 preferably about 1200 psi, a modulus at 200 % from about 1300 psi to about 1500 psi, more preferably about 1400 psi, and a modulus at 300 % from about 1700 psi to about

1900 psi, more preferably about 1800 psi.

The polyurethane preferably comprises formulants such as antiblocking agents, defoamers, surfactants, levelling agents and viscosity modifiers to assist in producing a coating that is substantially free of defects such as bubbles, craters and thin spots.

The polyurethane may also comprise a silane coupling agent to further promote adhesion to the glass. Preferred silane coupling agents include epoxysilanes, mercaptosilanes, and amino silanes. Gamma glycidoxypopyl trimethoxysilane, aminoethylaminopopyl trimethoxysilane, styrylaminoasilane and a product sold under the name of Silquest A-187 by Crompton Corporation USA are particularly preferred coupling agents. When a silane coupling agent is employed, it is preferable that it is prehydrolysed to form silanol monomers, silanol dimers, silanol oligomers or mixtures thereof. The prehydrolysed coupling agents are believed to be more effective at promoting adhesion.

When preparing the glass laminate, the polyurethane may be applied where necessary to effect bonding between the layer(s) within the laminate. For example, the polyurethane may be applied on one or both sides of the glass sheets and/or the interlayer(s). More preferably, the polyurethane is applied to both sides of the interlayer(s). Preferably, the polyurethane is applied as a substantially continuous coating on one or both sides of the glass sheets and/or the interlayer(s).

Application of the polyurethane onto the glass sheets or interlayer is typically achieved by some form of coating means. Preferred coating means include spray, rotogravure and reverse roll coating. The polyurethane is preferably applied to both sides of the interlayer, the process of which has been found to be advantageously robust and simple. In this regard, a polyurethane is typically applied to one surface of the interlayer, using coating means well known in the art, as part of continuous coating process. Once coated, the interlayer is transferred to a means for drying or curing the polyurethane, the interlayer is then returned for coating on the reverse side and after this coat has been dried or cured, the interlayer is typically coiled and stored ready for use in a subsequent lamination process.

- 7 -

Drying or curing of the polyurethane during the coating process may be achieved by means well known in the art. Preferred drying or curing means include transferring the coated interlayer through an oven or past an application of hot air. During the drying or curing process, it is important that the interlayer does not undergo a significant degree of orientation or downgauging. In order to minimise orientation or downgauging, a balance needs to be obtained between the temperature that interlayer reaches during the process and the force applied to the interlayer by the transferring means during its transit through the process. Preferably, the drying or curing process should not cause more than about 10 % orientation of the interlayer, more preferably not more than about 5% orientation, most preferably not more than about 2 % orientation. Where an oven is used to dry or cure the polyurethane, the temperature in the oven preferably ranges from about 30 °C to about 70 °C, more preferably from about 40 °C to about 60 °C.

Preferably, the polyurethane is applied to the glass sheets and/or interlayer(s) at a thickness ranging from about 5 GMS (dry) to about 70 GMS (dry), more preferably from about 10 GMS (dry) to about 50 GMS (dry), most preferably about 10 GMS (dry).

Where the polyurethane is applied to the interlayer, the coated self-adhering interlayer is typically coiled or stacked in the form of die-cut blanks for convenient storage and subsequent use in a lamination process. In this case, blocking can sometimes cause a problem with the subsequent separation of the sheets of interlayer. Blocking is a form of autoadhesion whereby the cured coating sticks to itself. It is a phenomenon that increases with storage time, pressure and temperature. If blocking of the coiled or stacked self-adhering interlayer occurs, a suitable interleaf material, such as a polyethylene sheet, can be used to assist in parting of the sheets of interlayer. Alternatively, the polyurethane can be formulated to avoid or at least minimise blocking. In this regard, the polyurethane preferably comprises an antiblocking or dulling agent. For example, a silica (SiO₂) agent, such as Stahl WD2863 sold by Stahl USA, or polyurethane micro-beads, such as Stahl WT13-485 sold by Stahl USA, may be incorporated in the formulation to minimise blocking. Provided that there is no net detrimental effect on the adhesive characteristics of the polyurethane, any additive(s) commonly used in polyurethane formulations that may

- 8 -

contribute to blocking can also simply be left out of the formulation. For example, it may be preferable to exclude tackifiers from the polyurethane formulation.

Advantageously, the coated self-adhering interlayer can be used as a "drop in" substitute
5 for a conventional PVB interlayer, and processed using standard laminating techniques. However, it is preferable to use a slightly higher temperature during pressing of the laminate than that used in a conventional PVB laminating process. In particular, temperatures ranging from about 10 to 20 °C higher than that used in a conventional PVB
10 laminating process are preferred. The slightly higher temperatures allow the polyurethane to flow more readily, which in turn facilitates the removal of entrapped air between layers and ensures a satisfactory bond between the interlayer(s) and the glass is obtained.

Once the layers of the laminate have been physically assembled, pressing of the laminate typically involves moving the composite through an oven operating at a temperature
15 approximately 120 °C between nip rollers exerting a pressure of around 1000 psi. This process softens the polyurethane which in turn initiates bonding between the layers. The nip rollers facilitate removal of entrapped air in the laminated sheet. The laminated sheets so formed are then transferred to an autoclave which typically operates at a temperature of about 135 °C and a pressure of about 1150 psi. Autoclaving is used to complete the
20 bonding process between the layers and typically takes around 4.5 hours.

The autoclave process provides uniform pressure that allows for further pressing of the coated PVC interlayer intimately against the glass. The uniform pressure prevents flow
25 differentials that could otherwise stretch the interlayer and distort a decorative pattern or effect. Alternatively, a further step may be employed which comprises placing the assembled laminated structure in a sealed vacuum bag to further minimise air bubbles from being entrapped within the laminate. The "bagged" structure is pre-pressed and/or pre-evacuated before the assembly is placed in an autoclave and heated to approximately 135
30 °C for 4.5 hours.

As mentioned above, PVC can readily be calendered to a range of thickness. Accordingly,

calendered PVC film is a particularly preferred form of PVC for use as an interlayer in accordance with the present invention. Interlayers comprising calendered PVC coated with a polyurethane can advantageously be used as a "drop-in" replacement for PVB interlayers in standard laminating processes. Accordingly, no additional manufacturing costs or modifications are required to conventional laminated glazing equipment.

The PVC used in the manufacture of the interlayers is preferably substantially free of impurities and sensitisers such as initiators, which can lead to excessive degradation and yellowing of the interlayer and can have a detrimental effect on the long term heat stability of the interlayer. In order to minimise the presence of these impurities and sensitisers, the PVC used in the manufacture of the interlayers is preferably made by suspension polymerisation.

Interfacial adhesion is a critical performance property for the glass-interlayer-glass system. During cutting of laminated glass, any delamination becomes highly visible and manifests itself as a type of 'edge whitening'. Even with good interfacial adhesion the tendency for edge delamination will increase with increasing hardness of the interlayer. In order to reduce the tendency for edge whitening, PVC used in the manufacture of the interlayer is preferably plasticised PVC.

Some plasticisers used for plasticising PVC are renowned for their tendency to migrate to the surface of a PVC product over time. Excessive migration of plasticiser from the PVC of the interlayer used in accordance with the present invention may interfere with the interfacial bonding between the interlayer and the layers of glass. Accordingly, if present, plasticiser(s) used in the PVC of the interlayer preferably have a low tendency for migration. Preferred low migration plasticisers include di-octyl adipate and polymeric plasticisers such as polyphthalates and polyadipates.

The PVC used for the interlayer preferably has a number average molecular weight of at least 40,000 Dalton and a plasticiser concentration ranging from about 60 phr to about 230 phr. An interlayer comprising plasticised PVC preferably has a hardness ranging from

about 15 BSS (British Standard Softness) to about 50 BBS.

Techniques to colour or decorate PVC are well known in the art. PVC may be pigmented to provide for solid (opaque) colours or novel pigments may be used to give special effects. Examples of novel pigments can include so called "interference pigments" or "colour variable pigments" which provide a differing colour perception depending on the viewing angle. PVC is also well suited to printing which can be used to apply an endless array of decorative effects. The decorative effect can be of any suitable colour or finish for example, marble, gloss, satin, translucent or flat. An interlayer comprising PVC can for example be produced with a black finish on one side and a white finish on the other side. This has the advantage of being able to use a single interlayer to produce a laminated glass which appears black on one side and white on the other.

An interlayer used in accordance with the present invention is preferably pigmented, coloured, printed or decorated in some way. Particularly preferred interlayers are those which are pigmented and which have had a printing design applied to one or both surfaces.

An interlayer used in accordance with the present invention preferably contains between about 2 to 4 phr of a primary heat stabilizer including an organometallic compound, such as salts of alkaline metals including barium, calcium, tin, and zinc. The primary heat stabilizer preferably includes a zinc salt of an organic acid or a barium, calcium or tin salt of an organic acid, or mixtures thereof. The primary heat stabilizer more preferably includes from about 1.6 to about 4.0 % atomic zinc as a zinc salt of an organic acid.

To maximise the long term thermal and colour stability of the interlayer, the primary heat stabilizer also preferably includes from about 2.0 weight % to about 4.0 weight % phosphorus in the form of a phosphite. A preferred phosphite is triphenyl phosphite.

The interlayer may also contain a UV stabilizer and/or a UV inhibitor to protect the PVC against the damaging effects of UV radiation.

- 11 -

The interlayer may also contain dyes, inorganic fillers, inorganic pigments, interference pigments and optical brighteners.

The interlayer in accordance with the present invention may also comprise a PVC
5 copolymer such as polyvinylchloride co-vinylacetate. In this case, the vinylacetate content of the copolymer preferably ranges from about 15 molar % to about 40 molar %. Suitable examples of vinyl chloride/vinyl acetate copolymers that are commercially available include MPR-TSN from Nissin Chemicals, Nitta-Gun, Japan, which is a copolymer of 87
10 400; and UCAR VYHD from Union Carbide, which is a copolymer of 86 molar % vinyl chloride and 14 molar % vinyl acetate with a degree of polymerisation of 220.

Examples

15 Tests commonly used to evaluate glass laminates include the following:

Impact Test

The most critical test for grade-A laminated safety glass to comply with is the impact test as defined in AS2208-96. In this test a 46 kg bag of lead shot is swung through a
20 pendulum arc at a vertical sheet of glass from a pendulum drop height of 300 mm. Essentially if the glass panel cracks into small pieces and deforms but is still held together in a safe manner, it is deemed to pass the test. It must retain the shards of annealed glass in an accident, limiting lacerations.

25 Adhesion by Pummel Test

The pummel test is used to measure interlayer adhesion to the glass. Twelve inch square glass laminates are placed in a freezer compartment at -18°C for at least two hours. After removal from the freezer, the laminates are placed on a metal substrate and hit repeatedly with a 450 g hammer to break the glass. All broken glass un-adhered to the interlayer is
30 then removed. The amount of glass left adhered to the interlayer is visually compared with a set of standards of known pummel scale and a pummel value for each sample is assigned.

Pummel values range from 0 (no adhesion; no glass adhered) to 10 (high adhesion, 100% of the glass adhered). In assessing the sample, it is to be noted that the bulk of the glass can break off but if the interfacial adhesion is good there will be a thin layer of glass remaining adhered to the interlayer.

5

Bake Test

In the bake test, a sample is heated in an oven at successively higher temperatures (82, 93, 104, 116, 127, 138 and 149°C) with 2 hours exposure to each temperature stage. The total bubble count is recorded.

10

Heat Yellowing

Heat stability is determined by monitoring the yellowing rate of laminated samples in one or more temperature controlled ovens. After measuring the initial yellowness index (YI) using a handheld colorimeter, samples are placed in one of five ovens set at various temperatures (65, 80, 100, 120 and 150°C). The samples are removed from the ovens at regular intervals and the YI is measured. The various time intervals used were as follows: 500 hours for the oven at 65°C, 250 hours for the oven at 80°C, 48 hours for the oven at 100°C, 24 hours for the oven at 120°C, and 4 hours for the oven at 150°C.

15

20

Boil Test

In the boil test (which is a part of AS2208), the samples, in triplicate are immersed vertically on edge in water at 66°C for 3 minutes and then quickly transferred to and immersed similarly in boiling water. The samples are kept in the boiling water for 2 hours, then removed and examined for bubbles and other defects. This test can highlight the presence of entrapped air. The degree of adhesion, yellowness and haze are also measured after the samples are subjected to a boil test.

25

Lap Shear Test

In this shear test, a lap joint containing the interlayer is subjected to tensile testing in a uniaxial manner. The shear modulus and shear strength of the laminate are recorded.

30

Interfacial Shear Test

The adhesive strength between the layers of a laminated glass is best characterised by measuring its interfacial shear strength. This parameter is assessed in accordance with ASTM F521 – Test Method B.

5

Samples tested

Unless otherwise indicated, the laminates tested were prepared using the following components:

10 Glass Sheet used

Commercially available float glass having a thickness of 3 mm.

PVC interlayers used

15 19BSS (British Standard Softness) PVC sheet of 0.19 mm thickness comprising 35 parts per hundred (phr) of DOP (dioctyl phthalate - also known as DEHP).

45BSS PVC sheet of 0.41 mm thickness comprising 60 parts per hundred (phr) of DOP (dioctyl phthalate - also known as DEHP).

20 Polyurethane coating used

A waterborne polyester polyurethane sold as RU41-347 by Stahl USA, Massachusetts, USA. In some instances the polyurethane was modified with a silane or siloxane coupling agent. In this case, Silquest A-187 siloxane sold by Crompton Corporation, South Charleston USA, or Stahl Siloxane sold by Stahl USA, Massachusetts, USA, was used
25 where indicated.

Coating application details

Coatings were applied to both sides of the PVC substrate using a #10 wire bar (or draw-down bar). Only one coat was applied in each case. The final coating thickness is a 8
30 GSM (dry) coat which approximates to a coating thickness of about 8 microns. The coating was cured in an oven at about 40 °C for about 12 hrs.

Lamination procedure followed

Unless otherwise indicated, the float glass sheets were first cleaned thoroughly with isopropyl alcohol. A coated self-adhering interlayer was placed on top of a glass sheet and a further glass sheet was placed on top of the interlayer to make a sandwich type structure.

- 5 The corners of the structure were held together with adhesive tape and the edges of the structure were then covered with perforated cellophane tape such that air could escape but the interlayer would be prevented from oozing out under pressure. The structure was then wrapped in a cotton blanket and placed in a flexible heat-proof plastic bag. Prior to sealing bag with temperature resistant putty tape one end of a tube was inserted into the bag with
- 10 the other end remaining outside of the bag. The bag was then placed in autoclave and the loose end of the tube was connected to a vacuum pump which applied a negative pressure of 5-15 psi to the bag assembly. The bag assembly was then heated in an autoclave for 4.5 hrs at 138 °C. The bag assembly was removed from the autoclave and allow to cool to room temperature before the laminate was unpacked.

15

Test Results

Table 1: Interfacial shear test results of laminates prepared using a 45 BSS PVC interlayer.

20

<i>Sample No.</i>	<i>Shear Strength*</i>
1	423 psi
2	345 psi
3	443 psi
4	378 psi
5	414 psi
6	404 psi
7	452 psi
8	383 psi

*Average shear strength is 405 psi.

- 15 -

Table 2: Test results of laminates prepared using a 45 BSS PVC interlayer.

Test	Result
Pendulum impact (AS2208-96)	pass*
Pummel test	8-9 (pass; good adhesion)
Bake test	no defects (pass)
Boil test (AS2208)	no defects (pass)
Shear Strength (ASTM F521-method B)	405 psi

*The glass shatters and splinters as a result of the impact but the broken glass is safely retained by the PVC interlayer.

5

Table 3: Test results of laminates that were also subjected to oven aging for 25 days at 82 °C after lamination.

Sample No.	Sample Description	Shear Strength (psi)	Standard Deviation
Reference sample	PVB interlayer	755	174
9	PVC(45 BSS) + RU41-347	996	112
10	PVC(45 BSS) + RU41-347 + 1.5% Silquest silane	807	50
11	PVC(45 BSS) + RU41-347 + 1.5% Stahl siloxane	829	183
12	PVC(19 BSS) + RU41-347	860	174
13	PVC(19 BSS) + RU41-347 + 1.5% Silquest silane	1296	178
14	PVC(19 BSS) + RU41-347 + 1.5% Stahl siloxane	927	51

- 10 The results shown in Table 3 indicate that a substantial increase in interfacial bond strength can be obtained by oven aging the samples after the autoclave step.

**This Page is Inserted by IFW Indexing and Scanning
Operations and is not part of the Official Record**

BEST AVAILABLE IMAGES

Defective images within this document are accurate representations of the original documents submitted by the applicant.

Defects in the images include but are not limited to the items checked:

- ☐ BLACK BORDERS
- ☐ IMAGE CUT OFF AT TOP, BOTTOM OR SIDES
- ☐ FADED TEXT OR DRAWING
- ☐ BLURRED OR ILLEGIBLE TEXT OR DRAWING
- ☐ SKEWED/SLANTED IMAGES
- ☐ COLOR OR BLACK AND WHITE PHOTOGRAPHS
- ☐ GRAY SCALE DOCUMENTS
- ☒ LINES OR MARKS ON ORIGINAL DOCUMENT
- ☐ REFERENCE(S) OR EXHIBIT(S) SUBMITTED ARE POOR QUALITY
- ☐ OTHER: _____

IMAGES ARE BEST AVAILABLE COPY.

As rescanning these documents will not correct the image problems checked, please do not report these problems to the IFW Image Problem Mailbox.